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Soil Sample Dissolution Development by Ultrawave Digester, Followed by Isotopic Separation and Analysis

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for August 2011 to March 2013

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14. ABSTRACT

The need for rapid nuclear assessment of radioactive contamination is being upgraded in readiness for reactor failure such as the recent Fukushima disaster or nuclear terrorism events. Current methods require large field samples and lengthy sample preparation and analysis time. The purpose of this study was to examine the efficiency of microwave-enhanced sample preparation methods for rapid sample decomposition, allowing chemical separations and spectrometry analyses. A unique new laboratory and potentially field-portable microwave unit, Milestone’s UltraWAVE Single Reaction Chamber microwave, has just become available for the accelerated mineral acid digestion of samples. Matrices to be tested included biological, botanical, sediment, and soil standards and standard reference materials (SRMs) and/or reference standards of each of these types and were chosen as examples. SRMs were obtained from the National Institute of Standards and Technology (Gaithersburg, MD). Each SRM contains metals known to be radioactive and of nuclear interest. Milestone’s UltraWAVE Single Reaction Chamber technology is suited for both laboratory and field analyses, since various matrices can be more rapidly acid digested simultaneously, reducing sample preparation time and increasing analysis efficiency. Plutonium, uranium, americium, and thorium were analyzed, along with other transition and rare earth metals, utilizing inductively coupled plasma-mass spectrometry and/or alpha spectrometry, following digestion. For validation of the microwave protocol, radioactive contaminated samples of a Rocky Flats soil were chosen. This SRM was analyzed at Wright-Patterson Air Force Base in collaboration with Applied Isotope Technologies and Duquesne University. Rocky Flats soil originated from the Rocky Flats National Laboratory in Colorado, where the soil and surrounding vegetation were heavily contaminated from a series of industrial accidents that released radionuclides to the environment.

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TABLE OF CONTENTS

Section **Page**

LIST OF FIGURES

Page

LIST OF TABLES

Page

1.0 SUMMARY

The need for rapid nuclear assessment of radioactive contamination is being upgraded in readiness for reactor failure such as the recent Fukushima disaster or nuclear terrorism events. Current methods require large field samples and lengthy sample preparation and analysis time. The purpose of this study was to examine the efficiency of microwave-enhanced sample preparation methods for rapid sample decomposition, allowing chemical separations and spectrometry analyses. A unique new laboratory and potentially field portable microwave unit, Milestone's UltraWAVE Single Reaction Chamber microwave, has just become available for the accelerated mineral acid digestion of samples.

Matrices to be tested included biological, botanical, sediment, and soil standards and standard reference materials (SRMs) and/or reference standards of each of these types and were chosen as examples. SRMs were obtained from the National Institute of Standards and Technology (Gaithersburg, MD). Each SRM contains metals known to be radioactive and of nuclear interest. Milestone's UltraWAVE Single Reaction Chamber technology is suited for both laboratory and field analyses, since various matrices can be more rapidly acid digested simultaneously, reducing sample preparation time and increasing analysis efficiency. Plutonium, uranium, americium, and thorium were analyzed, along with other transition and rare earth metals, utilizing inductively coupled plasma-mass spectrometry and/or alpha spectrometry, following digestion. For validation of the microwave protocol, radioactive contaminated samples of a Rocky Flats soil were chosen. This SRM was analyzed at Wright-Patterson Air Force Base in collaboration with Applied Isotope Technologies and Duquesne University. Rocky Flats soil originated from the Rocky Flats National Laboratory in Colorado, where the soil and surrounding vegetation were heavily contaminated from a series of industrial accidents that released radionuclides to the environment.

2.0 PART I: MICROWAVE DECOMPOSITION

2.1 Background

In traditional dissolution chemistry, the open vessel hot plate digestion method had been used since it was relatively easy to perform. The major drawbacks of this method were the time required and the possible contamination from the atmosphere or contamination to the surroundings (e.g., radioactive material), since the reaction was performed in an open system. A more advanced method for digestion is the utilization of a high temperature and pressure microwave decomposition system. Microwave decomposition systems have advanced to the point where there are units capable of breaking down various matrix constituents and releasing the analytes of interest for instrumental analysis. Previous work by Kingston and Jassie showed that 140ºC was required to break carbohydrate bonds, 150ºC for protein bonds, and 160ºC for lipid bonds by utilizing high temperature microwaves in conjunction with nitric acid (HNO_3) [1]. In microwave-induced energy systems, there are two heating principles that cause the digestion mixtures to heat. Ionic conductance occurs when there is an electromagnetic field applied; the ions in solution move with the sign of the field and the friction that results from the resistance to the movement causes heating. At the same time, the second mechanism of dipole rotation occurs, which is the alignment of the molecules by an oscillating electromagnetic field; the rapid shift in

dipole alignment causes heating to occur through molecular friction of the matrix and sample directly.

The use of microwaves for digestions was developed because there was a justifiable need for "better" chemistry. The chemistry changes when one shifts the paradigm of sample preparation from hot plate digestion to closed microwave-enhanced digestion systems. The fundamental chemistry that allows for this transfer is the Arrhenius equation as seen in Eq. 1. The integrated form is:

$$In\frac{K_2}{K_1} = \frac{E_a}{R}\left(\frac{1}{T_1} - \frac{1}{T_2}\right) \quad (1)$$

where K_1 and K_2 are the rate constants at temperatures T_1 and T_2, E_a is the activation energy, and R is the gas constant [2]. Eq. 1 shows the relationship between temperature and the rate of the reaction. In a hot plate digestion, the temperature of the digestion can only occur at the boiling point of the mineral acid that is being used. This is due to the reaction occurring at atmospheric pressure. Microwave digestion is carried out at elevated pressures, which increase the boiling point of the mineral acid and alter the chemistry of the reactions. The increased temperature of the mineral acid causes the reaction rate to increase dramatically. A secondary effect of the reaction rate increasing is that the time the digestion takes to complete shortens because the chemistry is occurring with faster kinetics. One example is the use of HNO_3; it is a strong oxidizing acid that increases its oxidizing potential with temperature elevation. The use of induced microwave energy with HNO_3 is an example of an ideal situation enabling efficiency enhancement based on Eq. 1.

One important acid to discuss is hydrofluoric acid (HF), since it is required for samples that contain siliceous compounds. HF produces an "opening-out" reaction breaking the silica oxygen bond. HF by itself is a non-oxidizing acid that is utilized primarily for this reaction and its complexing ability. There are two drawbacks to utilizing HF in digestion procedures: (1) it has the attribute of forming insoluble or sparingly soluble precipitates and (2) it also complexes with alkaline earth, lanthanide, and actinide elements. HF is a hazardous acid to work with and it is highly toxic.

In this evaluation and validation, the actinides are of particular importance, so care must be taken when utilizing HF in the dissolution because complete digestion is required; insoluble fluorides are not desired. There are several ways to alleviate the problem of using HF. One method is to substitute the anion of the desired analytes by evaporating the HF from the solution and then dissolve as single lone drop of liquid and converting the anions of the cations to nitrates. In the post-decomposition workup of the samples, HF has a lower boiling point than HNO_3; therefore, it can be removed by evaporation. Another method is to utilize boric acid (H_3BO_3) in the digestion procedure. To better understand the reaction involved with the acid decomposition and workup, Eqs. 2, 3, and 4 show the basic reaction principles of utilizing HF and H_3BO_3 when the matrices contain silicates:

$$SiO_2 + 6HF \rightarrow H_2SiF_6 + 2H_2O \quad (2)$$

$$H_3BO_3 + 3HF \rightarrow HBF_3(OH) + 2H_2O \quad (3)$$

$$HBF_3(OH) + HF \rightarrow [H]BF_4 + H_2O \quad (4)$$

Eq. 2 shows the silica oxygen bond in silica dissociated by HF, a reaction that is unique to HF. Without this reaction, complete chemical decomposition would not be achieved. Eq. 3 symbolizes the complexation of fluoride ions by H_3BO_3. Eq. 4 depicts the chemical reactions between H_3BO_3 and HF to form a volatile product that boils at 130ºC and assists in the liberation of the HF from the reaction mixture by chemically removing the HF as a volatile species. To overcome the slow kinetics of reaction 3, the HF in solution is reacted with an excess of H_3BO_3. This combination can be useful in dissolution of various matrices containing silicates and when elements of interest readily form insoluble fluoride compounds.

Along with the HNO_3, HF, and H_3BO_3, the addition of hydrochloric acid (HCl) is beneficial since numerous carbonates, peroxides, and hydroxides are soluble in HCl. HCl is most commonly used in conjunction with HNO_3, forming what is commonly known as aqua regia. Depending upon the elements that are contained in the matrix of the samples or standard reference materials (SRMs), various amounts of HCl can be added. There are several particular cases where HCl is a necessary addition to the digestion mixture. HCl is needed if the matrix has a high content of iron (Fe) because HCl can complex with Fe(II) and Fe(III). Also, if the quantification of antimony or silver is required, HCl stabilizes the dissolution of the two elements [3].

The last addition to the digestion mixture for this study was hydrogen peroxide (H_2O_2). H_2O_2 with a concentration of 30% can react explosively with organic material. For this study, H_2O_2 was utilized solely for the sediment and soil at Applied Isotope Technologies (AIT) and with inductively coupled plasma mass spectrometry (ICP-MS) analysis. H_2O_2 was not used with the Rocky Flats soil because it was deemed that it was not needed to achieve complete digestion of the material. H_2O_2 has the unique ability to "regenerate" or re-oxidize HNO_3 during the dissolution process. Eqs. 5 and 6 show this unique ability by suppressing the formation of nitrous oxide, which ultimately forms more HNO_3 since there are hydrogen ions present from the digestion:

$$NO_x \rightarrow NO_3^- \quad (5)$$

$$NO_3^- + H^+ \rightarrow HNO_3 \quad (6)$$

In optimizing the digestion, the proper ratio and concentration of acids must be achieved, with the correct microwave energy induction temperature heating profile. After the stoichiometry is determined, the correct ratio of constituents for the digestion mixture begins by analyzing the elements to be digested in the specific sample matrix. At the kick-off meeting, four "demonstration SRMs" were recommended for analysis: SRM 1570a, Trace Elements in Spinach Leaves; SRM 1646a, Estuarine Sediment; SRM 2709a, San Joaquin Soil; and SRM 1566b, Oyster Tissue [4-7]. The "validation SRM" that was chosen and analyzed at Wright-Patterson Air Force Base (WPAFB) was SRM 4353A, Rocky Flats Soil Number 2 [8]. Rocky Flats soil originated from the Rocky Flats National Laboratory in Colorado, where the soil and surrounding vegetation were heavily contaminated from a series of industrial accidents that released radionuclides to the environment [9].

Discussion of the first four SRMs for demonstration of the new microwave equipment to prepare for the validation at WPAFB is presented here. By reviewing the stoichiometry of both matrices and analyte elements in the certificates of each material, a stoichiometric quantity of HF, needed for complete digestion, was determined. The spinach and oyster material should have minimal amount of silicates, since silicates are normally associated with earthen materials, such

as soil and rocks. The certificates for spinach and oyster do not contain any information about silicon (Si) content [4,7]. When analyzing the estuarine sediment and soil certificate, certified values for the mass percent of Si were listed. The sediment and soil SRMs have approximately 40% and 30% Si, respectively [5,6]. Using those percentages, stoichiometric calculations were performed to determine a starting volume of HF. The calculations reveal that the minimum volumes needed for the soil and sediment were 0.35 and 0.13 mL, respectively. These calculations also take into account the appropriate amount of material needed for each digestion. Each certificate contains a usage statement that indicates to the user how much of the material is required to achieve the listed certified value based on homogeneity and particle size of the SRM [4-7].

The SRMs analyzed were chosen because each material contains certified values for elements that are known to be of nuclear interest. These SRMs also permit the assessment of each component of the method and analysis such as the microwave efficiency, the ICP-MS analysis, and the radioassays. The spinach provides a botanical reference, the oyster a biological reference, and the soil and sediment represent their respective matrices. The elements of interest to the personnel at the Air Force research site at WPAFB include barium (Ba), strontium (Sr), thorium (Th), uranium (U), neptunium (Np), plutonium (Pu), americium (Am), and curium. Select subsets of these elements were analyzed at WPAFB in collaboration with AIT and Duquesne University personnel in accordance with the Statement of Work (SOW). The analyses of isotopic Th, U, Pu, and Am were conducted by alpha spectrometry after sample dissolution by the newly developed UltraWAVE single reaction chamber (SRC) microwave digestion system (Milestone, Inc., Shelton, CT). Minimum detectable activity for these radionuclides is below 0.05 pCi.

2.2 Research and Assessment

2.2.1 Instrumental. The sample preparation for this study was completed using a newly developed laboratory microwave system (UltraWAVE) equipped with temperature and pressure feedback control. Figure 1 shows the microwave system and a brief schematic of how it operates [10]. This device is accurate in temperature sensing and control to within ± 2.0°C of set temperatures and automatically adjusts the microwave field output power to achieve preset temperature profiles of programmed protocols [10]. Extremely high temperature and pressure (300°C and 190 bar) conditions can be maintained for extended periods, so nearly any sample can be decomposed with mineral acids. Since all samples are held under the same conditions, different sample types can be digested in the same run. A maximum of 15 samples can be digested in a batch, which takes less than 45 minutes to complete. Glass, quartz, and/or TFM-polytetrafluoroethylene (PTFE) tubes can be used as sample vials for digestion. The vessels used for digestion were TFM-PTFE vessels obtained from Milestone Inc. Samples were digested in batches of 15. The 15-position rotor was used at AIT and the 5-position rotor was used at WPAFB, as the size of the sample was increased from 250 mg to 1.00 g. The caps were cleaned by soaking overnight in 1% ARISTAR® ULTRA HNO_3 (VWR, Radnor, PA) in deionized water.

The initial four SRMs decomposition protocols were developed and optimized at AIT in Pittsburgh, PA. The UltraWAVE was then transported to WPAFB and the Rocky Flats contaminated soil SRM protocol was developed and optimized at WPAFB with WPAFB personnel.

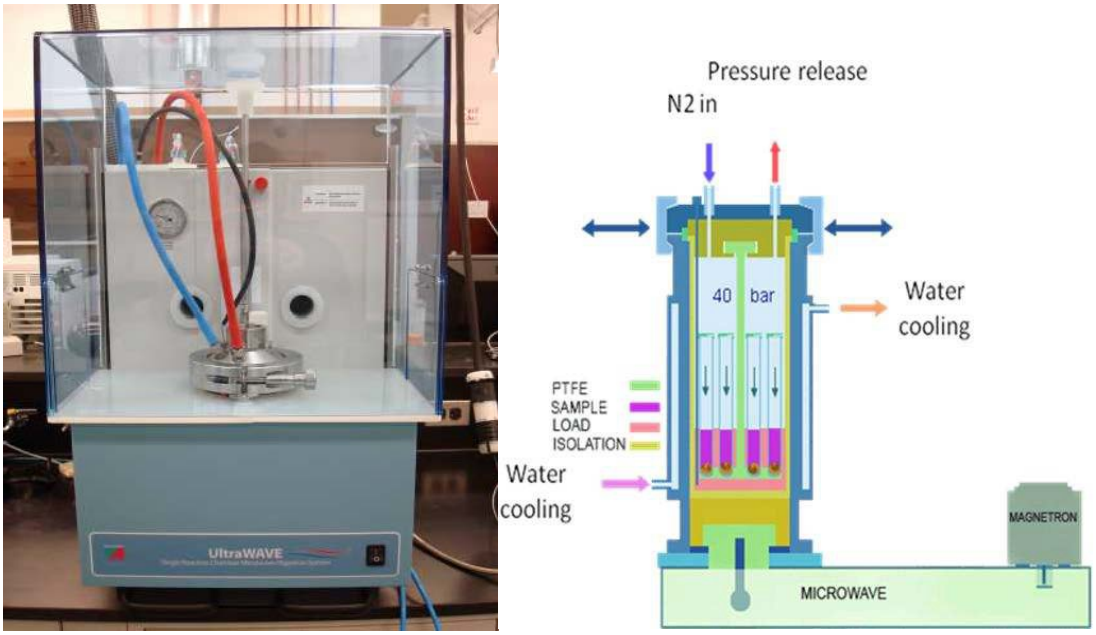

Figure 1. The Milestone UltraWAVE unit and SRC schematic. *The microwave easily fits on the bench top alongside its controller and its chiller on the floor. The instrument weighs less than 200 pounds, which is the limit for in-field transportability by military personnel. The chamber, as shown in the schematic, is charged with a non-reactive gas to a pressure of at least 40 bar. The entire jacket is water-cooled and the samples are immersed into a load solution that maximizes the microwave energy from the magnetron.*

The mass spectrometry analyses took place at AIT in Pittsburgh and were performed using an Agilent 7700 ICP-MS (Agilent Technologies, Santa Clara, CA). The Agilent 7700 ICP-MS has a collision cell, which uses hydrogen and helium gas for the elimination of polyatomic interferences. For all samples analyzed, samples were introduced into the Agilent 7700 ICP-MS with a Cetac ASX-520 autosampler (Omaha, NE) housed inside an ENC-500 anti-contamination enclosure. The pulse to analogue factor was determined on each day of analysis, and the tuning of the instrument was carried out using the Agilent ICP-MS tuning solution. The system was aspirated with 1% HNO_3 and 0.25% HCl for 30 minutes before tuning the instrument. The instrument operating conditions are illustrated in Table 1, along with an image of ICP-MS instrument in Figure 2.

Table 1. Optimized Settings for the ICP-MS for EPA Method 6020A in SRM Analysis[a]

Agilent 7700 ICP-MS Parameter	Method 6020A Analysis Setting
Radio frequency power	1550 W
Spray chamber temperature	2°C
Acquisition mode	Spectrum
Peak pattern	3 points/mass
Sample depth	8 mm
Carrier gas	0.9 L/min
Dilution gas	0.15 L/min
H_2 cell gas flow	4.5 mL/min
He cell gas flow	5.5 mL/min
Gas stabilization time	30 s
Integration time	0.1-1.0 s/mass
Replicates	4
Nebulizer pump speed	0.40 rps
Uptake time	30 s
Stabilization time	30 s

EPA = Environmental Protection Agency.
[a]Developed by Dr. Mizan Rahman – AIT.

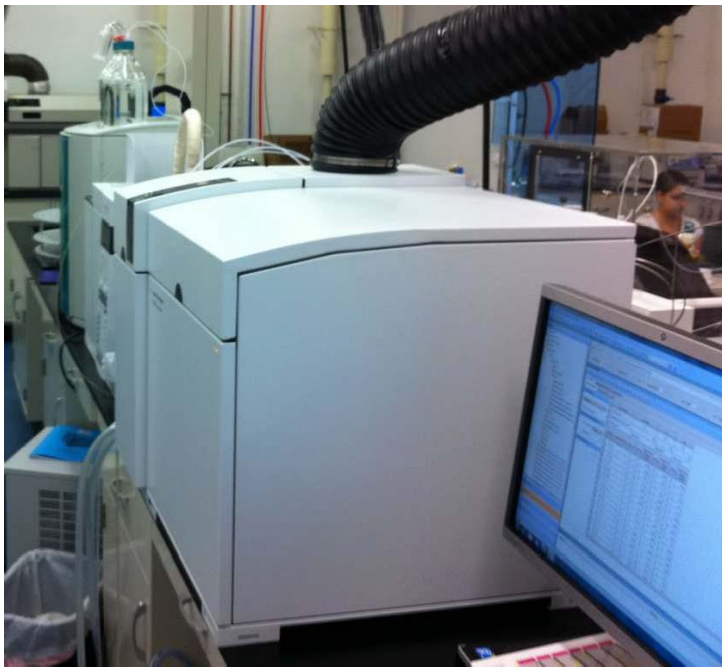

Figure 2. Image of ICP-MS at AIT that was utilized to analyze four of the SRMs for Part I of the study.

2.2.2 Standards and Chemicals. All reagents were of analytical or ultrapure grade. ARISTAR® ULTRA HNO_3, ARISTAR® ULTRA HCl, and ARISTAR® ULTRA H_2O were procured from VWR (Radnor, PA). Elemental standards A, B, and C were purchased from Inorganic Ventures (Christiansburg, VA). Agilent ICP-MS tuning mix consisting of 1 ng/mL lithium, cobalt, yttrium, cerium (Ce), and thallium in 2% (v:v) HNO_3 was used.

2.2.3 Sample Preparation Procedures. Four "demonstration SRMs" were acid digested during the study testing the equipment and developing methods and protocols on the UltraWAVE based on EPA Method 3052. These protocols were developed by Professor Kingston for the National Institute of Standards and Technology (NIST) and EPA and are performance-based methods requiring optimization for stoichiometry of matrix and analytes. The statistical protocol followed was to take three subsamples and then analyze them four times each. For each sample and subsample, 0.25 g or 0.50 g of sample was combined with 8 mL of ARISTAR® ULTRA HNO_3 and 0.25 mL of ARISTAR® ULTRA HCl, 1 mL of 30% H_2O_2 (for 1646a and 2709a), and 0.25 mL or 2.0 mL of concentrated HF as seen in Table 2. The samples were digested in the Milestone UltraWAVE microwave following EPA Method 3052. The parameters of Method 3052 include a 20-minute ramp to 225°C with a 20-minute hold at a minimum of 225°C [3]. After digestion, 0.25 g to 2.0 g of H_3BO_3 was added in each of the samples' digest based on the amount of HF used and then re-digested at 225°C for another 40 minutes. After this step, each sample was poured into a 50-mL polypropylene centrifuge tube and diluted to 20 mL using ARISTAR® ULTRA H_2O. The polypropylene tubes were weighed before and after the addition of sample digests. The samples were stored at room temperature until analyzed, usually within 1 day of digestion. Reagent blanks consisting of same amounts of concentrated HNO_3, HCl, H_2O_2, HF, and H_3BO_3 were also digested along with the samples.

Table 2. SRM Sample Preparation Guidelines

SRM	Sample (g)	HNO_3 (mL)	HCl (mL)	HF (mL)	H_2O_2 (mL)	H_3BO_3 (g)
1566b	0.250	8.00	0.250	0.250	0.00	0.250
1646a	0.500	8.00	0.250	2.000	1.00	2.000
2709a	0.250	8.00	0.250	2.000	1.00	2.000
1570a	0.250	8.00	0.250	0.250	0.00	0.250

2.2.4 Elemental Analysis by EPA Method 6020B. Total elemental analysis was performed using external calibration according to EPA Method 6020B [11]. Calibration curves were created for all the elements using the elemental standard solutions from Inorganic Ventures. The sample digests were further diluted to 1:50 with ARISTAR® ULTRA H_2O just before analysis with ICP-MS. Calibration standards were prepared by matrix-matching, which consisted of applying the appropriate percentage of microwave reagent blank to each calibration standard to match the acid content of each sample. The matrix-matched calibration standards were prepared at 0, 5, 10, 15, 20, and 25 ng/g. For each sequence of samples, three microwave blanks were prepared. Samples were loaded into the Cetac ASX-520 autosampler and analyzed. Analysis on the 7700 ICP-MS was performed in three analysis modes: hydrogen, helium, and no gas. A 30-second stabilization time was employed to equilibrate the presence or absence of gas when switching between analysis modes. The instrument was auto-tuned in all three analysis modes using the Agilent 1-ng/mL tune solution. Analyses were performed in spectrum mode with full quantification mode of three points per mass. The integration times for each element ranged from 0.1 to 1.0 seconds per point depending on the element's ionization energy. An Agilent Internal Standard solution was used at a concentration of 1 µg/mL in 1% ARISTAR® ULTRA HNO_3 and 0.5% ARISTAR® ULTRA HCl in ARISTAR® ULTRA H_2O. Between samples, the autosampler probe was washed in three solutions of 1% ARISTAR® ULTRA HNO_3 and 0.5%

ARISTAR® ULTRA HCl in ARISTAR® ULTRA H_2O for 30 seconds each and a nebulizer pump speed of 0.5 revolutions per second. The optimized parameters of the ICP-MS and autosampler are summarized in Table 1. After analysis, the data were exported from Agilent MassHunter Software to Microsoft Excel for data processing. The final samples (Rocky Flats Soil Number 2) were analyzed by alpha spectrometry at WPAFB instead of ICP-MS.

2.3 Results and Discussion

Microwave data can be exported to a computer that has the Milestone easyDOC software. The software allows the user to export the microwave run data to ensure that the instrument followed the settings and no problems occurred with the run. Figure 3 shows the graph the microwave generates as it runs the selected program. The figure shows the entire run of the microwave and the specific settings every 2 seconds. The temperature and maximum pressure profiles validate the programmed and actual conditions of the decomposition and power. They can be tracked to see what magnitude of power was required to maintain the set points for the method.

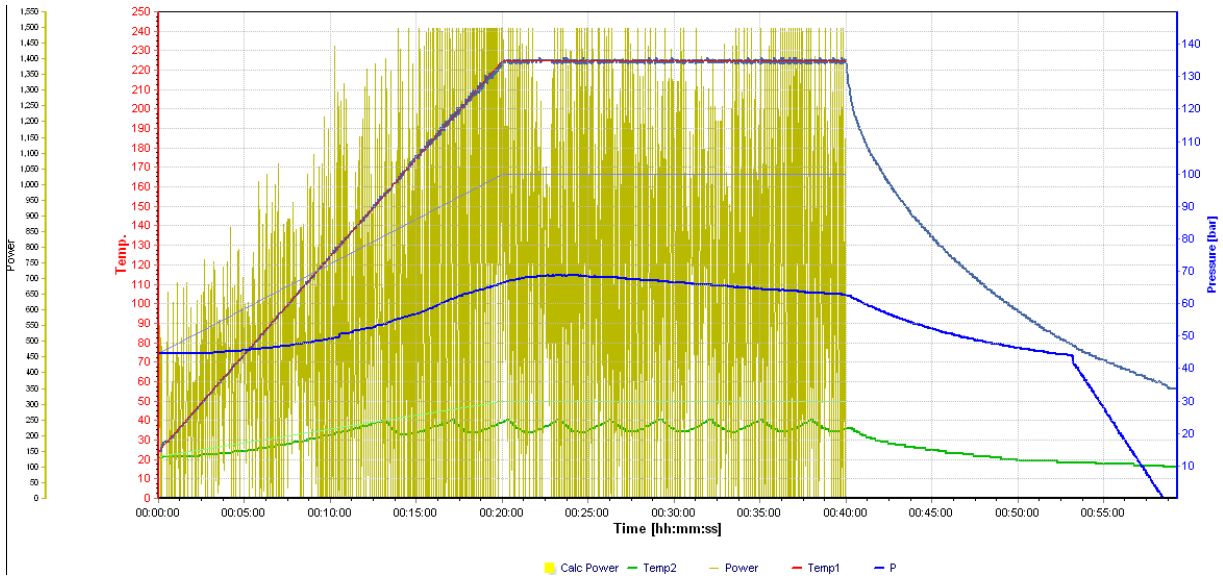

Figure 3. Microwave settings throughout the run that digested the four evaluation SRMs. *The graph shows multiple settings; green is temperature 2 (the vessel temperature), the thick blue line is the pressure of the system, the red line is temperature 1 (the reaction temperature), and the yellow is the power that is being applied by the magnetron to the reaction to follow the programmed settings of temperature 1.*

Following the data manipulation and calculations, the resulting concentrations in parts per billion (ppb) for the various elements of interest were calculated and summarized in Table 3. Duplicate error ratio (DER) and relative percent difference (RPD) were calculated to evaluate the agreement between the calculated concentrations and the certified or/and reference values. Uranium concentrations in the San Joaquin soil are in good agreement with the certified value, with a DER and a RPD of 1.02% and 2.84%, respectively, and show the element was released in solution after the dissolution process. Likewise, the calculated RPD for the Sr indicate an

acceptable recovery of the element and a successful dissolution. For Th and Ba, however, concentrations are lower than expected and indicate a possible loss during the analytical process. Results on biological matrices indicate better recoveries for Sr and Ba, indicating that some matrices are harder to digest, such as the soil and sediment SRMs, because they contain high Si content. High Si content compounds require opening out reactions to break the Si bonds as stated above. In addition, Th and Sr were not certified in SRM 1646a, and accuracy and precision cannot be determined without a certified standard. However, low results for Sr and Th were observed in 2709a, where they are certified, and additional research would be required to evaluate these biases. One method for Sr and U would be to apply IDMS using Sr-86, U-233, and or others (based on quantification objective) that would evaluate calibration curve bias and would be the preferred ICP-MS quantification of these isotopes and elements. The IDMS method would also compensate for Th "stickiness" and other challenging elements and isotopes.

Table 3. Results of Selected Elements from the Analysis of Four NIST SRMs Using EPA Methods 3052 and 6020B, Comparing Measured Concentrations to the Certified Reference Value (if available)

Element	Concentration	Uncertainty[a]	Certified Value	Uncertainty[a]	DER	RPD (%)
U						
1566b	3.70E+02	1.70E+01	2.55E+02	1.00E+00	**6.75**	**36.80**
1646a	1.84E+03	2.50E+01	2.00E+03			**8.28**
2709a	3.06E+03	7.10E+01	3.15E+03	5.00E+01	**1.02**	**2.84**
1570a	2.16E+02	3.00E+00	1.55E+02	2.30E+01	**2.63**	**32.90**
Sr						
1566b	7.15E+03	3.04E+02	6.80E+03	2.00E+02	**0.96**	**5.00**
1646a	4.48E+04	3.12E+03	6.80E+04			**41.20**
2709a	2.06E+05	2.61E+03	2.39E+05	6.00E+03	**4.98**	**14.60**
1570a	5.82E+04	2.97E+03	5.56E+04	8.00E+02	**0.83**	**4.51**
Th						
1566b	5.20E+01	4.00E+00	3.70E+01	4.00E+00	**2.65**	**33.70**
1646a	9.67E+02	2.10E+02	5.80E+03			**142.80**
2709a	3.31E+03	1.42E+02	1.09E+04	5.00E+01	**50.40**	**106.80**
1570a	4.40E+01	1.30E+01	4.80E+01	3.00E+00	**0.30**	**8.70**
Ba						
1566b	8.57E+03	3.68E+02	8.60E+03	3.00E+02	**0.07**	**0.40**
1646a	8.34E+04	1.05E+04	2.10E+05			**86.30**
2709a	6.19E+05	3.39E+03	9.79E+05	2.80E+04	**12.80**	**45.00**
1570a	7.00E+03	3.15E+02				

[a]Uncertainties are at 95% confidence interval with n = 12.

In addition, the lower recoveries of Th could be quantified if Th analyses are performed routinely, as Th adhering to glass and quartz equipment parts is a recognized phenomenon. Polyatomic interferences are not common in this region and are not known to be severe interferences with Th or Sr isotopes.

2.4 Training and Collaboration

The digestion protocols, including the acid combination, proved to be a viable method for the digestion of the four SRMs. Part of the SOW was the transfer of technology to WPAFB personnel also to train and transfer the technology. The first training was held at AIT and at Duquesne University to accomplish this goal the week of August 12, 2012. During this week of training, Dr. Aurelie Soreefan was provided a full 1-day subset of the American Chemical Society Sample Preparation course at Duquesne University and Microwave-Enhanced Chemistry, taught by Professor Kingston, the author of the course using EPA Method 3052 developed by Professor Kingston for NIST and EPA [12]. The next day of training was a 1-day practical application spent on four SRM samples and development of decomposition protocols using the Milestone UltraWAVE microwave unit. Part of that second day was devoted to training on the use of mass spectrometry based analysis by ICP-MS and practical application on the Agilent 7700 ICP-MS at AIT with training by Dr. Rahman. Method 6020B, the latest update of the Resource Conservation and Recovery Act, was taught. This new method approves the use of the latest collision cell technology with the ICP-MS [11].

On August 26, 2012, Professor Kingston and each of the project leaders traveled to Laval University and worked for a week coordinating the sample preparation methods and reviewing the applications with Professor Dominic Larivière, Marty Johnson, and Dr. Soreefan. Conference calls and project coordination for future ARSIIe [13] integration was planned and communicated.

While performing the decompositions at WPAFB, other members of the 711th Human Performance Wing were scheduled by their management to come to the laboratory where the new microwave equipment was being used. In discussions, a further need was identified for colleagues of the current project. Rapid decomposition of bomb fragments in medical applications was identified cooperation with military surgeons tending shrapnel victims. At present, they are spending up to 7 days decomposing single fragments and realized that the same methods being transferred to the radiation team could assist their effort to support the war fighter and provide required information to the medical staff. We left the application of microwave decomposition for this purpose and collaborations with the WPAFB personnel after discussions with these colleagues.

Specified in the SOW was the successful transfer of the microwave equipment and technology to WPAFB. The equipment and two week-long training and data-developing sessions were held on November 18-22, 2012, and December 16-22, 2012, at WPAFB. These technology transfers were successful, allowing WPAFB personnel to utilize this technology in their routine lab procedures. The transferred protocols now facilitate greater sample turnaround, replacing current hot plate digestion methods with more rapid and optimized microwave-enhanced digestion equipment and protocols.

Professor Kingston and Mr. Logan Miller demonstrated the ruggedness of the microwave equipment by transporting it by truck from Pittsburgh to WPAFB and setting it up and using it without having to modify it in any way. The unit is rugged enough to consider the next phase of taking this microwave equipment to the field and preparing samples under field conditions. However, this was beyond the scope of this project and is recommended for future work.

3.0 PART II: ISOTOPIC ANALYSIS

3.1 Validation of the Microwave System and Protocol Using Isotopic Analysis

WPAFB utilizes alpha spectrometry for the detection of alpha-emitting radionuclides. This technology was used for the validation of the microwave-enhanced protocols using the Rocky Flats Soil Number 2 SRM [8]. Validation data are attached and will be discussed in detail. In the future, the laboratory discussed the intent to perform quantitation of radionuclides of interest by ICP-MS analysis.

The analysis of the soil SRM was done in a series of two site visits to WPAFB. The primary focus of the site visits was to transfer and optimize the method using the Milestone UltraWAVE system with the sample decomposition protocol. In a second visit to WPAFB, the digestion solution was tested for its compatibility with the automated NorthStar Engineered Technologies Automated Radionuclide Separation System (Model ARSIIe – environmental version) for targeted radionuclides.

3.2 Validation and Research Using SRM 4353A (Rocky Flats Soil Number 2)

3.2.1 Instrumental. The sample preparation for the Rocky Flats Soil Number 2 SRM 4353A followed the same procedure as discussed above in Part I. The difference between the preparations was the sample size was increased to approximately 1 g of the SRM. Also, because alpha spectrometry was utilized for the analysis of radionuclides, NIST-traceable standard solutions (U-232, Pu-242, Th-229, and Am-243) were used as tracers for each element analyzed. Those tracers were added to the TFM-PTFE tubes prior to microwave irradiation and decomposition (UltraWAVE larger 5 rack tubes, each 25 mL capacity).

The alpha spectrometer utilized at WPAFB was a Canberra Alpha Analyst™ Integrated Alpha Spectrometer (Meriden, CT) [14]. The instrument is equipped with 24 vacuum chambers. This allowed for the analysis of 24 samples in a "batch mode" configuration. Alpha emitters of interest present in the SRM were Pu, U, Th, and Am. From 1-g soil sample digestate, four fractions (U, Pu, Th, and Am) were therefore each collected and converted to a solid source before counting in the alpha spectrometer chambers. Each Canberra Alpha Spectrometer chamber is equipped with recoil contamination protection, which helps to maintain consistent low backgrounds and extend the lifetime of the alpha detectors. The detectors in the alpha spectrometer are passivated implanted planar silicon, which surpasses the performance of silicon surface barrier detectors and diffused junction devices [14].

3.2.2 Sample Separation Using Vacuum Box. Following the digestion of the SRM, each sample and blank was evaporated to near dryness, then 5 mL of H_3BO_3 solution was added along with 3.5 M HNO_3 and 1 M aluminum nitrate. The digestate was then centrifuged for approximately 10 minutes at 3000 rpm to pellet any residual material. The supernatant was removed and saved for subsequent separation while the pellet (if any) was rinsed with 3.5 M HNO_3 and centrifuged again (supernatant removed and saved again.) The next step in the process is to separate each radionuclide. The following procedure was based on Eichrom Technologies, Inc. Analytical Procedure ACW16 VBS: Americium, Neptunium, Plutonium, Thorium, Curium, and Uranium in Water (with Vacuum Box System) [15]. The vacuum box system is assembled as instructed in ACW16 VBS upon which each supernatant is treated with 1.25 mL of 1.5 M

ascorbic acid. After 3 minutes, 1 mL of 3.5 M sodium nitrite was added to re-oxidize the oxidation state of Pu to Pu(IV). Each sample was then transferred to the vacuum box system funnels. The assembled vacuum box is shown below in Figure 4. To each sample vessel, 3 mL of 3 M HNO_3 was added to rinse each and transferred to the corresponding funnel; 5 mL of 3 M HNO_3 was then added to each funnel. The TEVA cartridge was separated from the TRU cartridge and then the elution steps were performed on each separate cartridge. The steps are summarized below. To elute Pu and Th from the TEVA cartridge, 10 mL of 3 M HNO_3 was added to each funnel, then 20 mL of 9 M HCL was added to elute Th. Each Th fraction was diluted to 45 mL with water and set aside for Ce fluoride (CeF_3) precipitation. Then, to the TEVA cartridge, 20 mL of 0.1 M HCl/0.05 M HF/0.03 M titanium chloride was added to elute the Pu. For the elution of Am from the TRU cartridge, 15 mL of 4 M HCL was added and collected, upon which each fraction was diluted to 30 mL with water and set aside for CeF_3 precipitation. Then, before eluting U from the TRU resin, 12 mL of 4 M HCl/0.2 M HF was added to strip any residual Th; 15 mL of 0.1 M ammonium bioxalate was added to elute U.

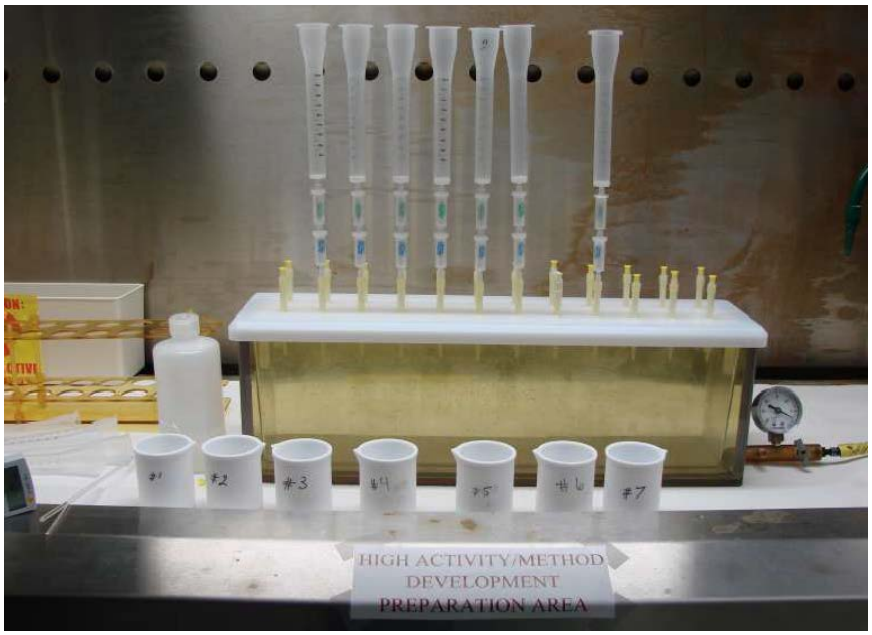

Figure 4. Vacuum box setup utilized for part of this study. *The two resins are connected together with the funnel attached to the top. The entire system is then connected to a vacuum line.*

3.2.3 Sample Separation Using ARSIIe. The sample preparation prior to separation with the ARSIIe is the same procedure as in the separation using the vacuum box. Prior to separation with the ARSIIe one additional step is required before the samples are loaded onto the ARSIIe. To the samples and the blank, 20 mg of Fe/ascorbic acid/sulfamic acid is added to the samples. The next step is to load the samples onto the ARSIIe. It is important that prior to loading onto the column, the Pu's oxidation state is reduced to Pu(III). Thus, the sodium nitrite addition implemented in the vacuum box protocol is not performed prior to loading the sample on the ARSIIe. The entire system is computer operated. The user chooses the correct separation protocol and then follows the on-screen instructions, which detail each step of the radionuclide separation. The ARSIIe system is shown below in Figure 5. A short abbreviated protocol is shown below in Figure 6.

Figure 5. NorthStar Engineered Technologies' ARSIIe. *The image above shows the four resins that are utilized labeled above in the image with the 1, 2, 3, and 4. To the left of the instrument are the tubes that contain the sample as well as rinse/eluting solutions. To the right are the collection tubes for each radionuclide.*

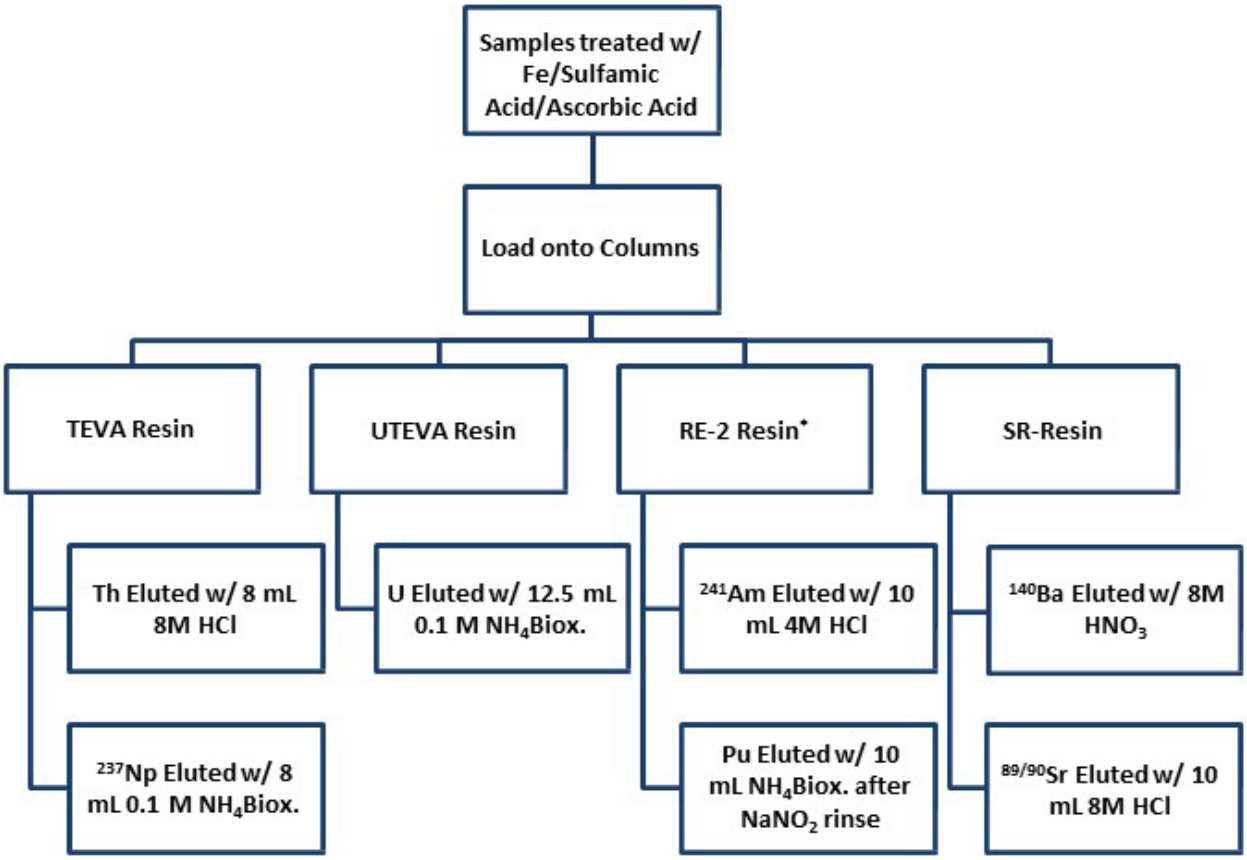

Figure 6. Flow chart representing the steps that the ARSIIe performs for the radionuclide separation. *The method shown above can separate and elute fractions of Th, ^{237}Np, U, ^{241}Am, Pu, Ba, and $^{89/90}$Sr. *RE-2 resin: a new Eichrom Technologies, Inc. proprietary resin.*

3.2.4 Preparation for Alpha Spectrometry. Once each fraction is eluted (either from vacuum box or ARSIIe), source preparation is implemented by a CeF_3 precipitation. To each fraction, 0.1 mL of Ce carrier was added, then 0.5 mL of titanium chloride was added to the U fractions only. H_2O_2 0.5 mL (30%) was added to the Pu fraction. One mL of concentrated HF was added to every Pu and U fraction, 3 mL HF to every Am fraction and 5 mL HF to every Th fraction. The solutions were mixed and left to sit for 30 minutes. Then they were filtered using 0.1-micron 25-mm Resolve filter (Eichrom Technologies, Inc., Lisle, IL). The filter was preconditioned with 80% ethanol and 2-3 mL water was added. The sample was filtered through and the sample vessel was rinsed with 5 mL water along with the filter being rinsed with 2-3 mL of water. Finally, 1-2 mL of 80% ethanol was added to displace the water and the sample and filter were dried under infrared lamps for a few minutes. Prior to counting, the filters were mounted onto stainless planchets using a glue stick and counted for 1000 minutes in the alpha spectrometer.

3.3 Results and Discussion

Figures 7 and 8 show the microwave data, which verify that the microwave applied the correct method that was developed and used for this project.

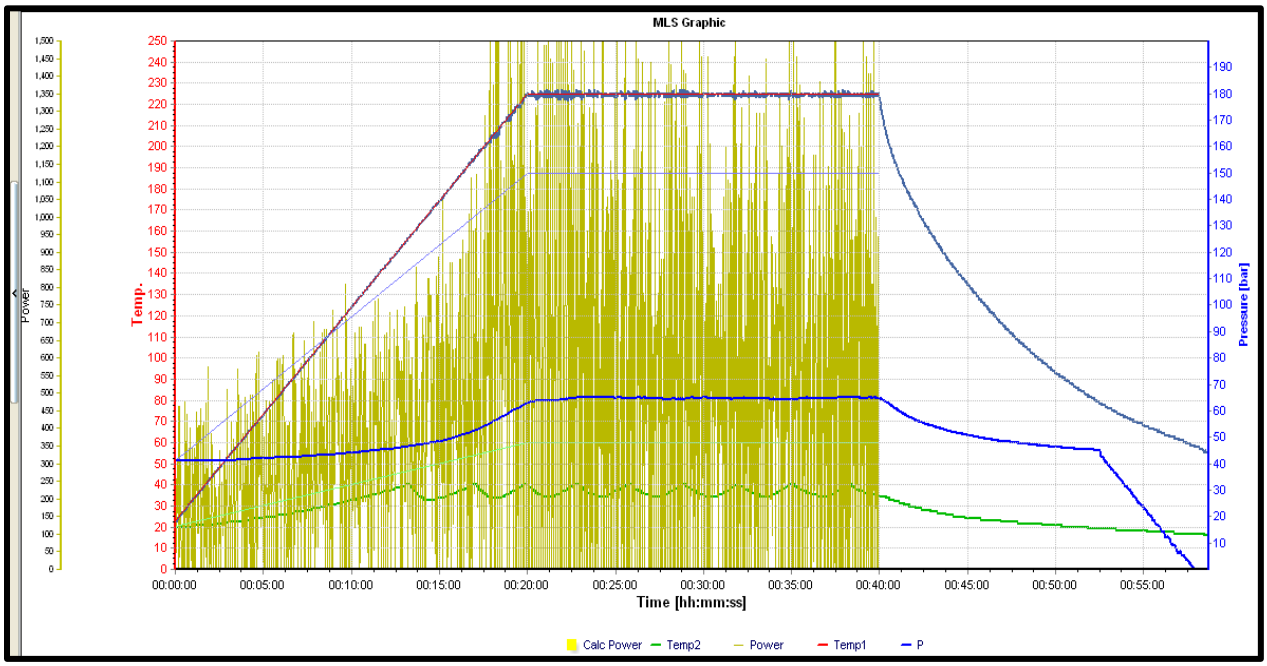

Figure 7. Microwave settings throughout the run that digested the SRM that was subsequently separated by the ARSIIe system. *The graph shows multiple settings; green is temperature two (the vessel temperature), the thick blue line is the pressure of the system, the red line is temperature one which (the reaction temperature), and the yellow is the power that is being applied by the magnetron to the reaction to follow the programmed settings of temperature 1.*

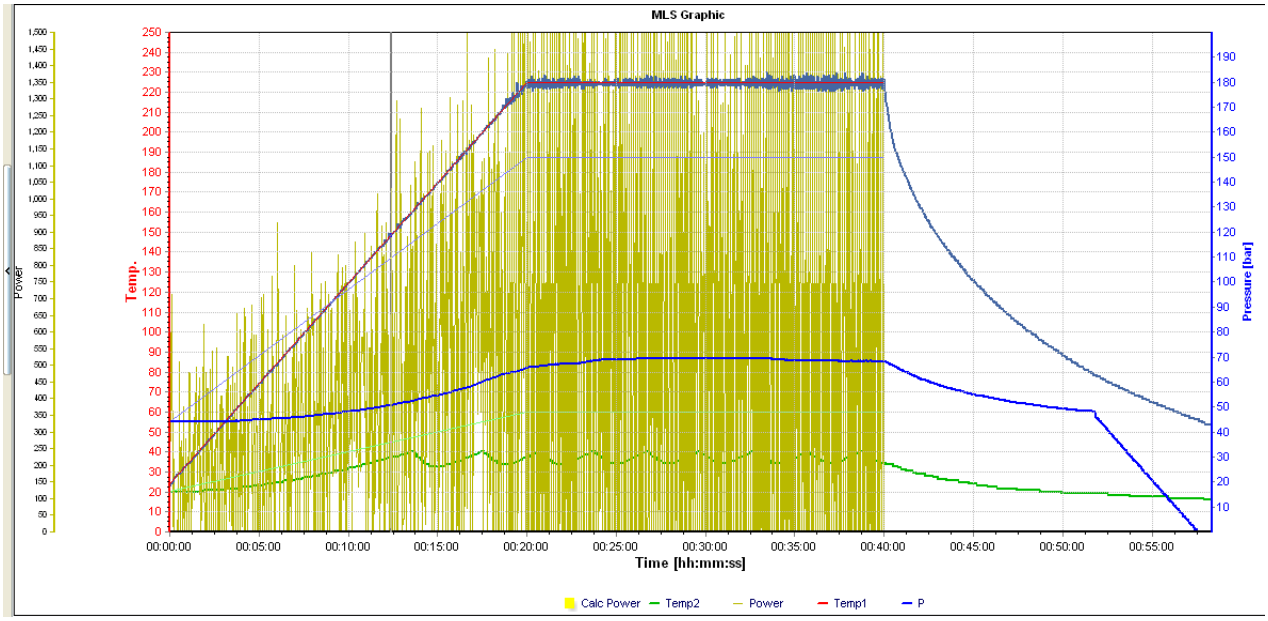

Figure 8. Microwave settings throughout the run that digested the SRM that was separated by the vacuum box system. *The graph shows multiple settings; green is temperature 2, which is the vessel temperature, the thick blue line is the pressure of the system, the red line is temperature 1 which is the reaction temperature, and the yellow is the power that is being applied by the magnetron to the reaction to follow the programmed settings of temperature 1.*

The alpha spectrometry data were obtained following either separation by the vacuum box system or by the ARSIIe and are summarized in Tables 4, 5, 6, and 7. Tables 4, 5, and 6 present data on the SRM, while Table 7 summarizes data from a separate performance evaluation material (Mixed Analyte Performance Evaluation Program [MAPEP]). The additional work on the latter was conducted to verify the recoveries of Am. DER (Eq. 7) and RPD (Eq. 8) were calculated, and the acceptable levels, per the Air Force, are ≤1.29 and ±25%, respectively. Both have to be in disagreement for the data to be considered unacceptable.

$$DER = \frac{|S - D|}{\sqrt{(2\sigma_S) + (2\sigma_D)}} \tag{7}$$

where S = first sample value (original)
D = second sample value (duplicate or reference)
$2\sigma_S$ = first sample 2σ uncertainty
$2\sigma_D$ = second sample 2σ ucertainty

And

$$RPD = \frac{|S - D|}{\left|\frac{(S + D)}{2}\right|} \tag{8}$$

where S = first sample value (original)
D = second sample value (duplicate or reference)

The original protocol developed for the digestion of the four SRMs at AIT in conjunction with Duquesne University utilized a sample size of 0.250 g. At WPAFB, the method was tested on a 1-g sample, a mass typically digested before alpha spectrometry measurement. In addition, the minimum detectable activity was decreased by a factor of four, which was necessary to detect Pu-239/240 present in the SRM.

Results from Tables 4 through 6 show that measured concentrations of U and Pu in the SRM were acceptable. Note that in Table 5, the tracer recoveries are for some samples over 100%. This was due to the slightly different geometries between the samples and calibration standards (filter vs. electroplated source).

Table 4. Samples Run on December 13, 2012, Using the Vacuum Box to Separate the Radionuclides

Analyses	Result (pCi/g)	Uncertainty (pCi/g)	Duplicate Result (pCi/g)	Duplicate Uncertainty (pCi/g)	DER	RPD
U1	**Tracer Recovery 94%**					
U-234	1.03E+00	1.34E-01	1.09E+00	8.11E-02	**0.40**	**5.84**
U-235	5.47E-02	3.19E-02	5.08E-02	1.43E-02	**0.11**	**7.39**
U-238	1.02E+00	1.34E-01	1.07E+00	8.11E-02	**0.32**	**4.78**
U2	**Tracer Recovery 82%**					
U-234	1.17E+00	1.54E-01	1.09E+00	8.11E-02	**0.45**	**6.90**
U-235	4.49E-02	3.14E-02	5.08E-02	1.43E-02	**0.17**	**12.30**
U-238	1.03E+00	1.44E-01	1.07E+00	8.11E-02	**0.24**	**3.81**
U3	**Tracer Recovery 96%**					
U-234	8.37E-01	1.56E-01	1.09E+00	8.11E-02	**1.45**	**26.40**
U-235	7.62E-02	5.26E-02	5.08E-02	1.43E-02	**0.47**	**40.00**
U-238	1.02E+00	1.73E-01	1.07E+00	8.11E-02	**0.26**	**4.78**
Pu1	**Tracer Recovery 95%**					
Pu-238	7.17E-03	1.16E-02	7.51E-03	1.11E-03	**0.03**	**4.63**
Pu-239/240	3.67E-01	7.31E-02	4.54E-01	4.86E-02	**0.99**	**21.20**
Pu2	**Tracer Recovery 93%**					
Pu-238	2.05E-03	8.92E-03	7.51E-03	1.11E-03	**0.61**	**114.00**
Pu-239/240	3.54E-01	7.31E-02	4.54E-01	4.86E-02	**1.14**	**24.80**
Pu3	**Tracer Recovery 96%**					
Pu-238	1.60E-03	1.58E-02	7.51E-03	1.11E-03	**0.37**	**130.00**
Pu-239/240	4.57E-01	1.09E-01	4.54E-01	4.86E-02	**0.03**	**0.66**

Note: Radionuclides of interest were uranium and plutonium. Red indicates the value lies outside of the reference values acceptance range.

Table 5. Samples Run on December 18, 2012, Using the Vacuum Box to Separate the Radionuclides

Analyses	Result (pCi/g)	Uncertainty (pCi/g)	Duplicate Result (pCi/g)	Duplicate Uncertainty (pCi/g)	DER	RPD
U4	**Tracer Recovery 108%**					
U-234	1.03E+00	1.23E-01	1.09E+00	8.11E-02	0.62	**8.80**
U-235	3.53E-02	2.39E-02	5.08E-02	1.43E-02	0.56	**36.00**
U-238	1.01E+00	1.24E-01	1.07E+00	8.11E-02	0.40	**5.77**
U5	**Tracer Recovery 93%**					
U-234	1.05E+00	1.33E-01	1.09E+00	8.11E-02	0.27	**3.92**
U-235	5.04E-02	3.01E-02	5.08E-02	1.43E-02	0.01	**0.79**
U-238	1.02E+00	1.31E-01	1.07E+00	8.11E-02	0.32	**4.78**
U6	**Tracer Recovery 105%**					
U-234	9.43E-01	1.20E-01	1.09E+00	8.11E-02	1.03	**14.60**
U-235	6.55E-02	3.33E-02	5.08E-02	1.43E-02	0.41	**25.30**
U-238	1.01E+00	1.25E-01	1.07E+00	8.11E-02	0.40	**5.77**
Pu4	**Tracer Recovery 101%**					
Pu-238	1.77E-02	1.75E-02	7.51E-03	1.11E-03	0.58	**80.80**
Pu-239/240	4.53E-01	7.97E-02	4.54E-01	4.86E-02	0.01	**0.22**
Pu5	**Tracer Recovery 95%**					
Pu-238	8.42E-04	8.28E-03	7.51E-03	1.11E-03	0.80	**160.00**
Pu-239/240	5.14E-01	8.41E-02	4.54E-01	4.86E-02	0.62	**12.40**
Pu6	**Tracer Recovery 102%**					
Pu-238	6.54E-03	1.06E-02	7.51E-03	1.11E-03	0.09	**13.80**
Pu-239/240	3.81E-01	7.11E-02	4.54E-01	4.86E-02	0.85	**17.50**
Th4	**Tracer Recovery 20%**					
Th-228	1.29E+03	1.59E+02	1.96E+00			**199.00**
Th-230	1.21E+00	3.03E-01	1.30E+00			**6.79**
Th-232	1.19E+00	3.00E-01	1.99E+00			**50.30**
Th5	**Tracer Recovery 16%**					
Th-228	1.51E+03	1.92E+02	1.96E+00			**199.00**
Th-230	1.68E+00	3.90E-01	1.30E+00			**25.90**
Th-232	1.41E+00	3.56E-01	1.99E+00			**34.10**
Th6	**Tracer Recovery 17%**					
Th-228	1.37E+03	1.72E+02	1.96E+00			**199.00**
Th-230	1.33E+00	3.30E-01	1.30E+00			**2.67**
Th-232	1.23E+00	3.19E-01	1.99E+00			**47.20**
Am4	**Tracer Recovery 11%**					
Am-241	1.53E+00	1.16E+00	6.76E-02			**183.00**
Am5	**Tracer Recovery 8.0%**					
Am-241	1.13E+00	1.28E+00	6.76E-02			**177.00**
Am6	**Tracer Recovery 55.8%**					
Am-241	3.57E-01	5.09E-01	6.76E-02			**136.00**

Note: Radionuclides of interest were U, Pu, Th, and Am. Red indicates the value lies outside of the reference values acceptance range.

Table 6. Samples Run Using the ARSIIe to Perform the Radionuclide Separation

Analyses	Result (pCi/g)	Uncertainty (pCi/g)	Duplicate Result (pCi/g)	Duplicate Uncertainty (pCi/g)	DER	RPD
RFSU1	**Tracer Recovery 99%**					
U-234	1.09E+00	1.34E-01	1.09E+00	8.11E-02	**0.01**	**0.20**
U-235	2.87E-02	2.22E-02	5.08E-02	1.43E-02	**0.84**	**55.60**
U-238	9.90E-01	1.26E-01	1.07E+00	8.11E-02	**0.53**	**7.80**
RFSU2	**Tracer Recovery 93%**					
U-234	1.06E+00	1.35E-01	1.09E+00	8.11E-02	**0.20**	**3.00**
U-235	3.36E-02	2.52E-02	5.08E-02	1.43E-02	**0.59**	**40.80**
U-238	9.80E-01	1.29E-01	1.07E+00	8.11E-02	**0.59**	**8.80**
RFSU3	**Tracer Recovery 106%**					
U-234	9.70E-01	1.20E-01	1.09E+00	8.11E-02	**0.84**	**11.80**
U-235	8.68E-02	3.75E-02	5.08E-02	1.43E-02	**0.90**	**52.30**
U-238	8.90E-01	1.15E-01	1.07E+00	8.11E-02	**1.28**	**18.40**
Pu1	**Tracer Recovery 98%**					
Pu-238	1.91E-03	8.33E-03	7.51E-03	1.11E-03	**0.67**	**119.00**
Pu-239/240	3.90E-01	7.40E-02	4.54E-01	4.86E-02	**0.72**	**15.20**
Pu2	**Tracer Recovery 62%**					
Pu-238	4.65E-03	1.27E-02	7.51E-03	1.11E-03	**0.22**	**47.00**
Pu-239/240	4.35E-01	9.84E-02	4.54E-01	4.86E-02	**0.17**	**4.27**
Pu3	**Tracer Recovery 9%**					
Pu-238	1.91E-03	8.35E-03	7.51E-03	1.11E-03	**0.66**	**119.00**
Pu-239/240	4.26E-01	7.75E-02	4.54E-01	4.86E-02	**0.31**	**6.36**
Th1	**Tracer Recovery 6.0%**					
Th-228	1.42E+03	3.04E+02	1.96E+00			**199.00**
Th-230	1.57E+00	6.29E-01	1.30E+00			**19.20**
Th-232	1.09E+00	5.21E-01	1.99E+00			**58.40**
Th2	**Tracer Recovery 7.0%**					
Th-228	1.16E+03	1.87E+02	1.96E+00			**199.00**
Th-230	1.76E+00	5.88E-01	1.30E+00			**30.40**
Th-232	1.28E+00	4.99E-01	1.99E+00			**43.40**
Th3	**Tracer Recovery 7.0%**					
Th-228	1.52E+03	2.84E+02	1.96E+00			**199.00**
Th-230	1.39E+00	5.92E-01	1.30E+00			**7.08**
Th-232	2.06E+00	6.48E-01	1.99E+00			**3.51**
Am1	**Tracer Recovery 45%**					
Am-241	1.26E-01	6.82E-02	6.76E-02			**60.40**
Am2	**Tracer Recovery 49%**					
Am-241	1.27E-01	6.45E-02	6.76E-02			**61.10**
Am3	**Tracer Recovery 38%**					
Am-241	6.55E-02	5.37E-02	6.76E-02			**3.11**

Note: Radionuclides of interest were U, Pu, Th, and Am. Red indicates the value lies outside of the reference values acceptance range.

Table 7. MAPEP Soil Sample Analyzed for Uranium, Plutonium, and Americium

Analyses	Result (pCi/g)	Uncertainty (pCi/g)	Duplicate Result (pCi/g)	Duplicate Uncertainty (pCi/g)	DER	RPD
MAS27-1-U	**Tracer Recovery 84%**					
U-234	5.70E+01	6.73E+00	6.03E+01	1.90E+00	0.47	5.63
U-235	6.34E-02	2.53E-02	5.33E-02	1.60E-03	0.40	17.30
U-238	2.45E+02	1.86E+01	2.63E+02	7.00E+00	0.91	7.09
MAS27-1-Pu	**Tracer Recovery 83%**					
Pu-238	1.04E+02	9.40E+00	1.06E+02	1.70E+00	0.23	2.10
Pu-239/240	1.25E+02	1.38E+01	1.34E+02	2.00E+00	0.61	6.63
MAS27-1-Am	**Tracer Recovery 65%**					
Am-241	1.04E+02	1.18E+01	1.11E+02	2.00E+00	0.56	6.19

Note: Red indicates the value lies outside of the reference values acceptance range.

Low recoveries were found for the Th tracer, which indicates the element was trapped or made insoluble during the dissolution process. Note that the Th-228 activities were biased high due to the presence of this isotope in the U-232 tracer solution. A simple “cleaning” step of the U-232 tracer solution would resolve this issue. Unsatisfactory results for Am in the SRM are due to the material having concentration levels close to the method detection limits. In addition, this specific SRM reference value was uncertified. The additional work performed on the MAPEP sample showed that the Am-241 results are in good agreement with the reference concentration value, thus demonstrating the dissolution method was satisfactory for Am.

4.0 CONCLUSIONS

This report is the final summary report of the training, technology development, technology transfer, and validation data. The objectives in the SOW were met and successfully accomplished as outlined for U, Pu, and Am. The method still needs optimization for Th. The equipment was transferred as were the equipment-specific protocols for microwave sample decomposition.

While not required in the SOW, it was observed that transportation in a vehicle, setup by laboratory personnel, and immediate application of the developed methods upon arrival in the laboratory were indications that the microwave equipment has the potential to be used as both a laboratory and field sample preparation device in homeland defense for reactor failure, thermonuclear device or dirty bomb monitoring, assessment, and actionable metrology instrumentation.

The AIT Team recommends one additional week of collaboration between Duquesne University and WPAFB personnel to accomplish one objective: additional data collection for a peer-reviewed paper and presentation. The successful method applications and data demonstrated that we have the methods and equipment for successful monitoring of critical radioisotopes in the laboratory setting and that the potential exists for future field applications.

The equipment chosen for this project could be field capable. The opportunity exists to extend this application to the field through a field demonstration involving sample preparation from raw contaminated soil and other environmental samples and production of actionable data in the field.

The equipment and each component of the equipment in this project were chosen with this future project goal in mind, and the AIT Team stands ready to collaborate when called upon.

5.0 REFERENCES

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LIST OF SYMBOLS, ABBREVIATIONS, AND ACRONYMS

AIT	Applied Isotope Technologies
Am	americium
Ba	barium
Ce	cerium
CeF_3	cerium fluoride
DER	duplicate error ratio
EPA	Environmental Protection Agency
Fe	iron
H_2O_2	hydrogen peroxide
H_3BO_3	boric acid
HCl	hydrochloric acid
HF	hydrofluoric acid
HNO_3	nitric acid
ICP-MS	inductively coupled plasma mass spectrometry
MAPEP	Mixed Analyte Performance Evaluation Program
NIST	National Institute of Standards and Technology
Np	neptunium
ppb	parts per billion
PTFE	polytetrafluoroethylene
Pu	plutonium
RPD	relative percent difference
Si	silicon
SOW	Statement of Work
Sr	strontium
SRC	single reaction chamber
SRM	standard reference material
Th	thorium
U	uranium
WPAFB	Wright-Patterson Air Force Base